

## Supporting information

*for*

### Tb<sup>3+</sup>/Sm<sup>3+</sup> co-doped double perovskite: synthesis, exfoliation and luminescence properties

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#### Experimental section

##### Preparation of double perovskite bulk

The double perovskite bulk was prepared by a solid-state reaction which is similar to literature.<sup>1</sup> Take the Tb<sup>3+</sup> doped double perovskite K(K<sub>1.5</sub>Tb<sub>0.5</sub>)Ta<sub>3</sub>O<sub>10</sub> (KKTTO) as an example. 5.528 g K<sub>2</sub>CO<sub>3</sub>, 2.815 g Tb<sub>4</sub>O<sub>7</sub>, 10.605 g Ta<sub>2</sub>O<sub>5</sub>, and 1.000 g NaF were mixed thoroughly in an agate mortar for 30 min, and heated in air at 800 °C for 6h. After cooling down, the bulk was re-grind and final calcined at 1200°C for 15h. The Tb<sup>3+</sup> and Sm<sup>3+</sup> co-doped KKTSTO samples were synthesized with similar method but replaced parts of the Tb<sub>4</sub>O<sub>7</sub> with Sm<sub>2</sub>O<sub>3</sub> to maintain Tb<sub>4</sub>O<sub>7</sub>:Sm<sub>2</sub>O<sub>3</sub> molar ratio of 1.0:0, 0.8:0.2, 0.6:0.4, and 0.5:0.5, respectively.

##### Exfoliation into nanosheet

10.0 g of the KKTSTO precursor was dispersed into 500 mL of 2 M HNO<sub>3</sub> for 3 days under continuous stirring. The HNO<sub>3</sub> solution was refreshed every 24 hours. After acid treatment, the protonated precursor was filtrated and washed with deionized water for several times until the pH~7, and then dried at room temperature.

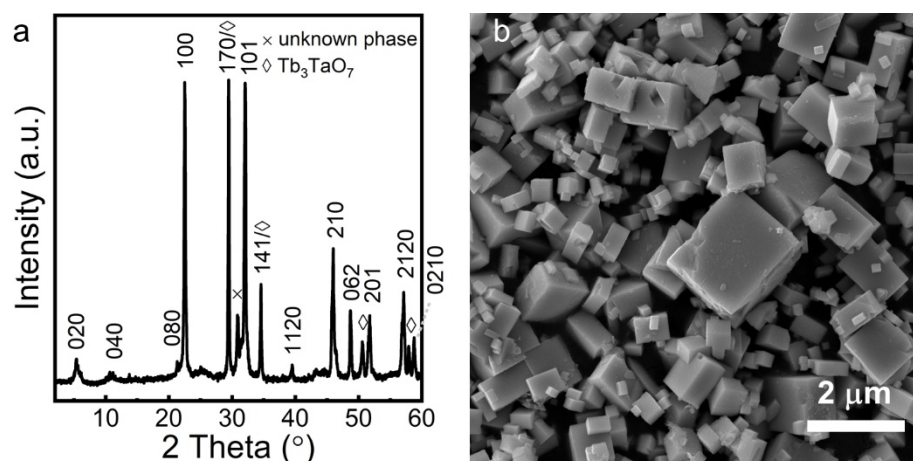
For exfoliation, 2.0 g of the protonated precursor was dispersed in 250 mL of

tetrabutylammonium hydroxide (TBAOH) aqueous solution with the molar ratio of  $H^+ : TBA^+ = 1:6$  and shook for 7 days under 180 rpm. The exfoliated nanosheet suspension was obtained by centrifuging the suspension at 3000 rpm for 30 min.

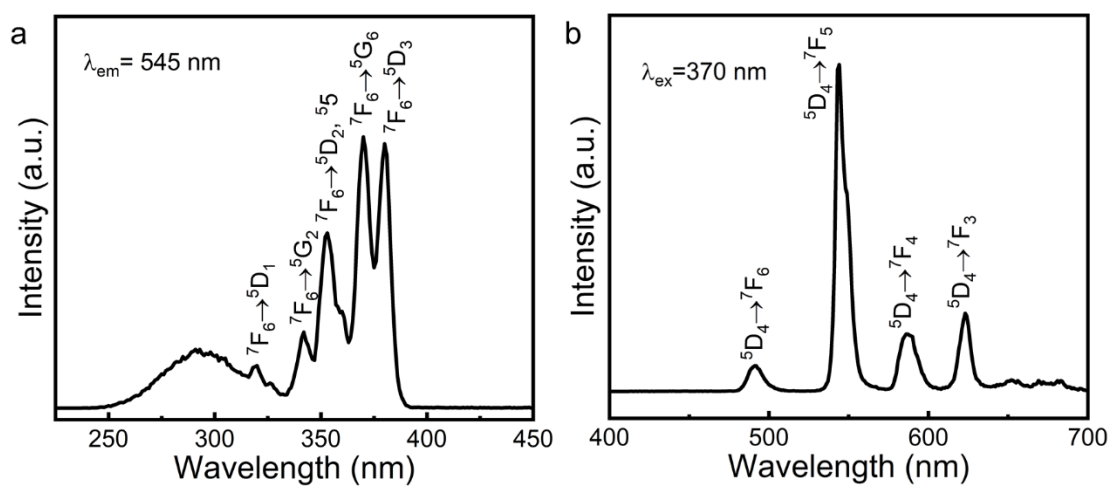
Silicon wafer was used as a substrate for atomic force microscopy (AFM) observation. Silicon wafer was first cleaned in a bath of methanol/HCl and then in concentrated  $H_2SO_4$ , and finally rinsed with deionized water. The silicon wafer was subsequently dipped into polyethyleneimine (PEI) solution for 30 min and then washed with deionized water. Nanosheets were deposited on the PEI modified wafer by dipping the wafer into the corresponding nanosheet suspension, followed by washing in deionized water and drying under the protection of  $N_2$  flow.

### **Characterization**

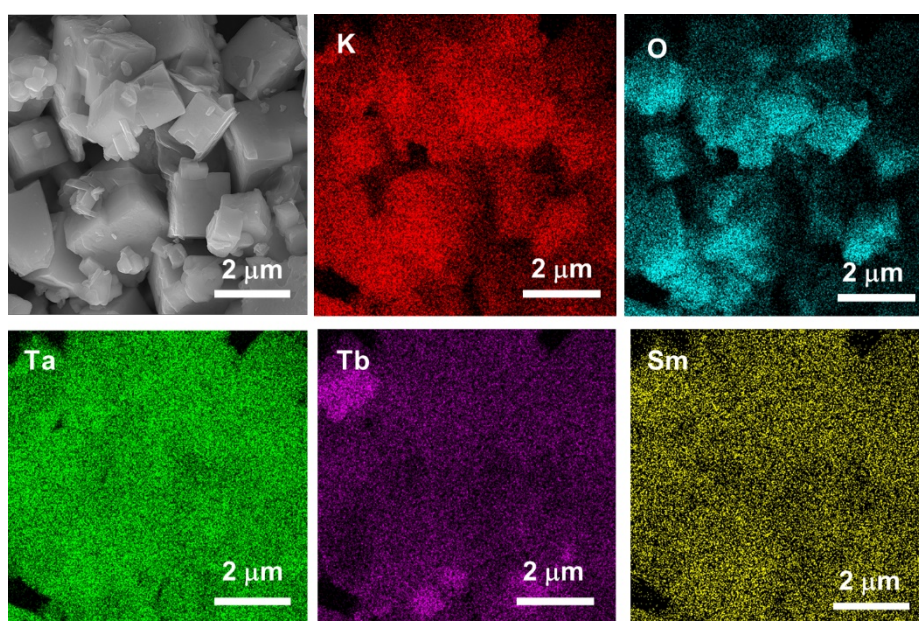
The X-ray diffraction (XRD) patterns were collected using a Rigaku Miniflex 600 diffractometer with  $Cu\ K\alpha$  radiation ( $\lambda = 0.15418\text{ nm}$ ). Scanning electron microscopy (SEM) was conducted using an FEI Helios Nanolab 600i microscope. Transmission electron microscopy (TEM) was performed on a Tecnai G2 F20 system. Atomic force microscopy (AFM) observations were carried out using a Seiko SPA 400 in tapping mode. Elemental compositions were determined by inductively coupled plasma-optical emission spectrometry (ICP-OES, Avio 200). The photoluminescence spectra and fluorescence decay curves were measured by using an Edinburgh FLS1000 photoluminescence spectrophotometer. The diffuse reflectance spectrum was measured by Shimadzu UV-2600 ultraviolet-visible spectrophotometer.



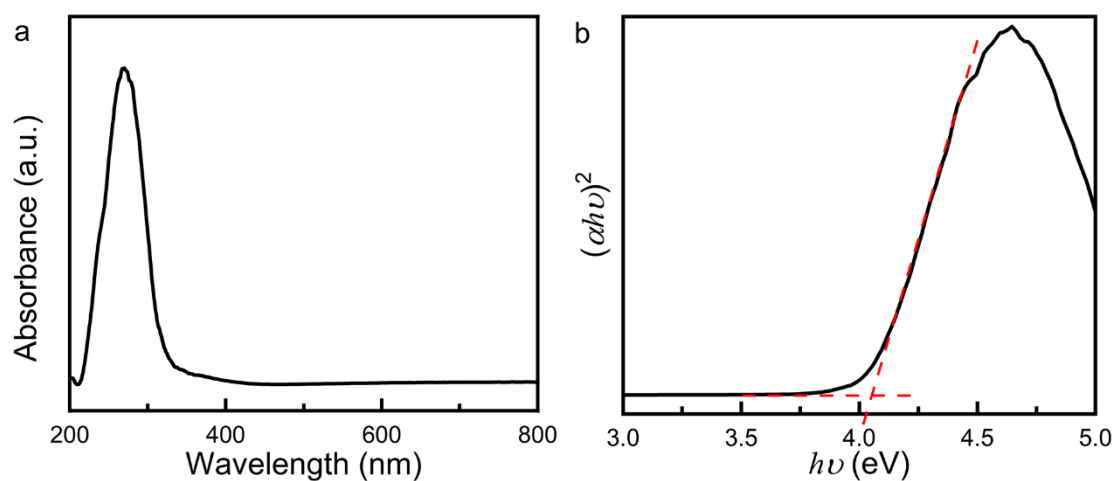
**Fig. S1.** (a) XRD pattern and (b) SEM image of KKTTO.



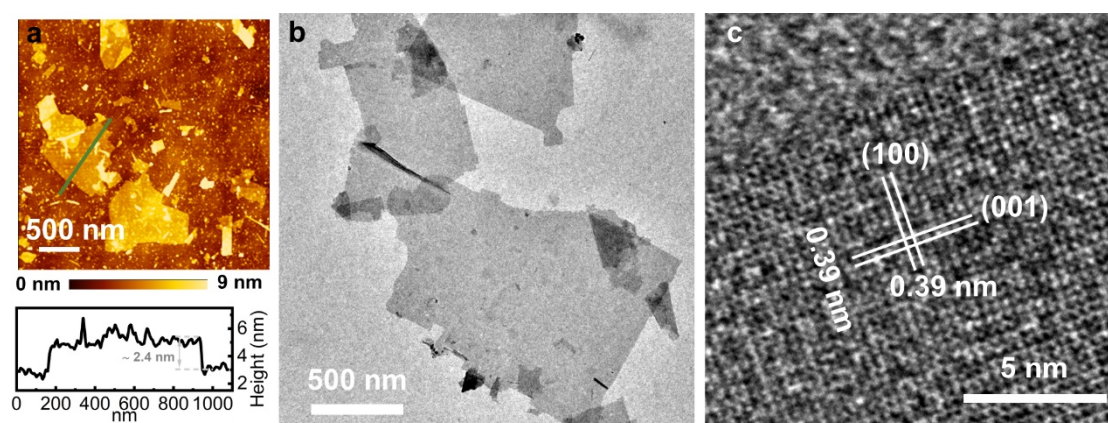
**Fig. S2.** Photoluminescence (a) excitation and (b) emission spectra of KKTTO.



**Fig. S3.** EDS elemental mapping images of the  $\text{KKT}_{0.8}\text{S}_{0.2}\text{TO}$ .



**Fig. S4.** (a) UV-vis diffused reflection spectra of the KKT<sub>0.8</sub>S<sub>0.2</sub>TO and (b) the plot of  $(\alpha h\nu)^2$  versus  $h\nu$  for the estimation of the bandgap.



**Fig. S5.** (a) AFM image and typical thickness, (b) TEM image, and (c) HRTEM image of KTTTO nanosheet.

## References

1. T. C. Ozawa, K. Fukuda, K. Akatsuka, Y. Ebina, T. Sasaki, K. Kurashima and K. Kosuda, *J. Phys. Chem. C*, 2008, **112**, 17115-17120.